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# *Indian Standard*

# BENTONITE FOR CERAMIC INDUSTRY— SPECIFICATION

भारतीय मानक

बेन्टोनाइट, सिरामिक उद्योग के लिए — विशिष्टि

( First Reprint NOVEMBER 1996 )

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**BUREAU OF INDIAN STANDARDS**
**MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG**
**NEW DELHI 110002**

*November* 1989 **Price Group 2**

Ceramicware Sectional Committee, CDC 27

## FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 26 December 1988, after the draft finalized by the Ceramicware Sectional Committee had been approved by the Chemical Division Council.

Bentonite is a hydrous silicate of alumina derived from volcanic ash with montnorillenite $(OH)_4Si_8Al_4O_{20}.nH_2O$ as its main mineral constituent. It is used as plasticizer in certain types of bodies to increase the drying time, fired strength and to reduce absorption. It is also used in high temperature cement mortars as a suspending agent in glazes enamel, etc.

This standard keeps in view the quality of bentonite available and as is being commercially exploited in the country by the ceramic industry as a whole.

The following Indian Standards on bentonite have already been published:

a) IS 6186 : 1971 Specification for bentonite

b) IS 10214 : 1982 Methods of sampling bentonite

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values ( *revised* )'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# AMENDMENT NO. 2 OCTOBER 2011
# TO
# IS 12621 : 1988 BENTONITE FOR CERAMIC INDUSTRY — SPECIFICATION

[*Page* 1, *Table* 1, *Sl No.* (xi), *col* 2] — Substitute 'Swelling power after 24 hours, ml' *for* 'Swelling power after 24 hours'.

(*Page* 3, *clause* **B-4.2.1**, *line* 2) — Substitute '**5.1.1**' *for* '**5.2.1**'.

(*Page* 4, *clause* **B-6.2.1**) — Substitute '**B-6.3.1** *Decomposition of the Sample*' *for* '**B-6.2.1** *Decomposition of the Sample*'.

(*Page* 8, *clause* **B-11.1**, *line* 1) — Substitute '**5.1.1**' *for* '**5.1.8**'.

(CHD 9)

Reprography Unit, BIS, New Delhi, India

# AMENDMENT NO. 1 JUNE 2007
# TO
# IS 12621 : 1988 BENTONITE FOR CERAMIC INDUSTRY — SPECIFICATION

(*Page* 1, *clause* 2) — Substitute the following for the existing clause

## 2 REFERENCES

The standards listed in Annex A contain provisions which through reference in this text, constitute provisions of this standard At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated in Annex A.'

(*Page* 1, *clause* **4.2**) — Insert the following at the end

**4.3** *BIS Certification Mark*

The bags may also be marked with the Standard Mark

**4.3.1** The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.'

(*Page* 2, *clause Reference to Annex* A) — Substitute '(Clause 2)' *for* (Clause 2.1)'.

(CHD 9)

Reprography Unit, BIS, New Delhi, India

# *Indian Standard*

# BENTONITE FOR CERAMIC INDUSTRY — SPECIFICATION

## 1 SCOPE

This standard prescribes the requirements and methods of sampling and test for bentonite of least swelling type for use in ceramic industry.

## 2 REFERENCES

The Indian Standards listed in Annex A are necessary adjuncts to this standard.

## 3 REQUIREMENTS

### 3.1 Description

The material shall be in the form of fine powder, free from dirt and other foreign matter.

**3.2** The material when tested according to the methods prescribed in the Annex B shall comply with the requirements specified in Table 1.

## 4 PACKING AND MARKING

### 4.1 Packing

The material shall be packed in jute bags or as agreed to between the purchaser and the manufacturer.

### 4.2 Marking

The bag shall be securely closed and marked indelibly with the following information:

a) Name of the material;
b) Grade of the material, that is 'Ceramic Grade';
c) Net mass;
d) Lot number to enable the batch of manufacture to be traced from records; and
e) Name of manufacturer or his registered trade-mark, if any.

**Table 1 Requirements For Bentonite**

| Sl No. | Characteristic | Requirement | Method of Teat, Ref to Cl. No. in Annex B |
|---|---|---|---|
| (1) | (2) | (3) | (4) |
| i) | Free moisture content at 105 ± 2°C, percent by mass, *Max* | 6 | B-2 |
| ii) | Residue on 106 micron IS Sieve (wet sieving), percent by mass, *Max* | Nil | B-3 |
| iii) | Grit content on 45 micron IS sieve, percent by mass, *Max* | 1.0 | B-4 |
| iv) | Loss on ignition, percent by mass | 8 to 12 | B-5 |
| v) | Silica (as $SiO_2$), percent by mass | 48 to 55 | B-6 |
| vi) | Alumina (as $Al_2O_3$), percent by mass | 18 to 28 | B-7 |
| vii) | Iron oxides (as $Fe_2O_3$), percent by mass, *Max* | 4 | B-8 |
| viii) | Titanium oxide (as $TiO_2$), percent by mass, *Max* | 3 | B-9 |
| ix) | Oxides of iron (as $Fe_2O_3$) and titanium (as $TiO_2$) together, percent by mass, *Min* | 6 | — |
| x) | Water of plasticity, percent by mass | 45 to 60 | B-10 |
| xi) | Swelling power after 24 hours | 15 to 20 | B-11 |
| xii) | Calcium oxide (as CaO), percent by mass, *Max* | 3 | B-12 |
| xiii) | Magnesium oxide (as MgO), percent by mass. *Max* | 3 | B-12 |
| xiv) | Oxides of calcium (as CaO) and magnesium (as MgO) together, percent by mass, *Max* | 5 | — |
| xv) | Viscosity at 30°C, centipoise, *Min* | 4.5 | B-13 |

NOTE — All tests except for Sl No. (i) shall be carried out on dry basis.

## 5 SAMPLING, NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

**5.1** Representative samples of the material shall be drawn from each lot as prescribed in **3** and **4** of IS 10214: 1982. The number of tests shall be carried out as given in **7** and **8** of IS 10214 : 1982.

**5.1.1** *Test Sample*

Take out 100 g of the representative sample, mixed thoroughly and dry at 105± 2°C to constant mass and use it for the purpose of chemical analysis.

**5.2 Criteria for Conformity**

The lot shall be declared as conforming to the requirements of this standard if all the test results meet the relevant requirements.

## ANNEX A

## ( *Clause* 2.1 )

### LIST OF REFERRED INDIAN STANDARDS

| *IS No.* | *Title* |
|---|---|
| IS 264 : 1976 | Specification for nitric acid ( *second revision* ) |
| IS 265: 1976 | Specification for hydrochloric acid ( *second revision* ) |
| IS 296 : 1986 | Specification for sodium carbonate, anhydrous (*second revision* ) |
| IS 1070 : 1977 | Specification for water for general laboratory use ( *second revision* ) |
| IS 1997 : 1982 | Specification for burettes ( *second revision* ) |
| IS 2080 : 1980 | Specification for stabilized hydrogen peroxide (*first revision* ) |
| IS 2837 ( Part 2 ) : 1977 | Specification for basins (*first revision* ) |
| IS 10214 : 1982 | Specification for methods of sampling bentonite |
| IS 10332 : 1982 | Specification for hydrofluoric acid, aqueous |

## ANNEX B

## ( *Clause* 3.2 )

### METHODS OF TEST FOR BENTONITE FOR CERAMIC INDUSTRY

### B-1 QUALITY OF REAGENTS

Unless otherwise specified, 'pure chemicals' and distilled water (*see* IS 1070 : 1977) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of the analysis.

### B-2 DETERMINATION OF MOISTURE

**B-2.1 Procedure**

Weigh accurately about 10 g of material in a tared porcelain dish, covered with a watch glass. Spread the material uniformally over the bottom of the dish. Keep it in an air oven maintained at 105 ± 2°C for three hours, cool in a desiccator and weigh. Repeat the process till constant weight is obtained.

NOTE — Avoid prolonged heating of the material at 110$^{0}$C and above to prevent gel formation.

**B-2.2 Calculation**

Moisture content, percent by mass

where

$M$ = mass in g of the sample taken for the test, and

$M_1$ = mass in g of the sample after drying.

### B-3 DETERMINATION OF RESIDUE ON SIEVE

**B-3.1 Equipment**

**B-3.1.1** *Test Sieve,* 106 micron IS sieve.

**B-3.1.2** *Shaking Machine*

**B-3.2 Procedure**

Weigh accurately about 10 g of the test sample ( *see* **5.1.1**) and place it in a 500-ml bottle and add gradually 350-ml of water. Shake the mixture thoroughly in a suitable shaking machine for a period of three hours. The mixture thus prepared shall be fluid and free from lumps.

**B-3.2.1** Sieve this mixture through 106 micron IS sieve, wash with a slow stream of water, dry it at 105 ± 2°C and weigh.

**B-3.2.2** The material shall be deemed to have passed the test if no residue left on the test sieve.

## B-4 GRIT CONTENT

### B-4.1 Equipment

**B-4.1.1** *Test Sieve,* 45 micron IS sieve.

**B-4.1.2** *Shaking Machine*

### B-4.2 Procedure

**B-4.2.1** Weigh accurately about 10 g of the test sample ( *see* **5.2.1** ) and place it in a 500-ml bottle and add 350ml of water slowly. Shake the mixture in a flask shaker for three hours. The mixture thus prepared should be fluid and free of lumps.

**B-4.2.2** Sieve this mixture through 45 micron IS sieve, wash with a slow stream of water, dry it at 105 ± 2°C and weigh.

**B-4.2.3** Report the material on the sieve as the percentage of the material taken.

### B-4.3 Calculation

Material retained on sieve,

percent by mass

where

$M_1$ = mass in g of the material retained on the sieve, and

$M$ = mass of the material taken for the test.

## B-5 DETERMINATION OF LOSS ON IGNITION

**B-5.1** Weigh accurately about 1 g of the test sample ( *see* **5.1.1**) in a platinum crucible. Heat gently for 15 minutes over a small flame till the bubbling ceases. Continue the ignition in a muffle furnace at 1 000 ± 25°C for 30 minutes, cool in a desiccator and weigh. Repeat the operation till constant weight is obtained.

### B-5.2 Calculation

Loss on ignition, percent by mass

where

$M_1$ = mass in g of the empty crucible,

$M_2$ = mass in g of the crucible with the material before ignition, and

$M_3$ = mass in g of the crucible with the material after ignition.

## B-6 DETERMINATION OF SILICA

### B-6.1 Outline of the Method

The sample is decomposed by fusion with sodium carbonate and extracted with acid. Silica is then determined gravimetrically by dehydration and baking followed by hydrofluorization. The residual silica in the filterate is determined photometrically using molybdenum blue method.

### B-6.2 Reagents

**B-6.2.1** *Sodium Carbonate,* anhydrous ( *see* IS 296 : 1986).

**B-6.2.2** *Dilute Hydrochloric Acid,* ( 1 : 1 ), ( 1 : 3 ) and ( 1 : 19 )( *v/v* ).

**B-6.2.3** *Dilute Sulphuric Acid,* 1 : 1 ( *v/v* ).

**B-6.2.4** *Hydroflouric Acid,* 40 percent ( *m/m* ) (*see* IS 10332 : 1982).

**B-6.2.5** *Potassium Pyrosulphate,* solid.

**B-6.2.6** *Ethylene Diamine Tetracetic Acid.* ( *EDTA* ) *Solution,* 0.025 M approx. Dissolve 9.31 g of sodium salt of ethylene diamine acid dihydrate ( $C_{10}H_{14}N_2N_2O_82H_2O$ ) in water and dilute to 1 litre.

**B-6.2.7** *Phenolphthalein Solution*

Dissolve 1 g of phenolphthalein in 100 ml of rectified spirit.

**B-6.2.8** *Dilute Ammonia Solution,* 1 : 3 ( *v/v* ).

**B-6.2.9** *Ferric Ammonium Sulphate Solution*

Dissolve 40.82 g of ferric ammonium sulphate $[ Fe NH_4 ( S0_4 )_2 . 12 H_20 ]$ in water containing 80 ml of dilute hydrochloric acid ( 1 : 1 ) and dilute to 500-ml in a volumetric flask.

**B-6.2.10** *Ammonium Molybdate Solution*

Dissolve 100 g of ammonium molybdate in water containing a few drops of dilute ammonia, filter if necessary and dilute to 1 litre.

**B-6.2.11** *Oxalic Acid Solution*

Dissolve 100 g of oxalic acid in water and dilute to 1 litre.

**B-6.2.12** *Ascorbic Acid Solution, 10 percent* **(** ***m/m*** **)**

Dissolve 10 g of the acid in 100 ml of water and filter, if necessary. Preserve in an amber coloured bottle. Teh solution, thus preserved may be used up to 5 days.

**B-6.2.13** *Standard Silica Solution,* 0.04 mg/ml.

a) *Stock solution*

Fuse 0.200 g of pure silica ( 998 percent) in 5 g of sodium carbonate in a platinum crucible. Dissolve in 300-ml of water containing 8 to 10 g of sodium hydroxide, using a polyethylene beaker. Cool and dilute with water in a volumetric flask to 500-ml. Store in a polyethylene bottle.

b) *Standard solution*

Prepare immediately before use, a standard solution containing 0.04 mg/ml by diluting the above stock solution appropriately.

### B-6.3 Procedure

**B-6.2.1** *Decomposition of the Sample*

**B-6.3.1.1** Weigh accurately about 1 g of the test sample (*see* **5.1.1** ) into a platinum crucible. Add 3 g of sodium carbonate. After mixing intimately, cover the dish with lid and heat over a Maker burner, slowly increasing the temperature with caution till the mixture fuses. Keep at this temperature for 10 minutes and finally heat till a clear melt is obtained. Remove from the burner and cool. Extract the melt in a porcelain dish first with water and then with ( 1:1 ) dilute hydrochloric acid.

**B-6.3.1.2** Add about 30 ml of ( 1 : 1 ) dilute hydrochloric acid. Cover the porcelain dish and allow to stand on a steam bath until disintegration of the melt is complete. Crush any large flakes with flattened end of a glass rod.

**B-6.3.2** *Gravimetric Determination of Insoluble Silica*

**B-6.3.2.1** Evaporate the solution in the porcelain dish to dryness and allow it to remain on steam bath for about an hour until the smell of hydrochloric acid is no longer detected. Transfer the dish to an air oven, maintained between 105 ± 2°C and bake for one hour. Cool the dish and add 20 ml of ( 1 : 1 ) dilute hydrochloric acid followed by 30 ml of hot water. Stir the mixture till solution of soluble salts appear to be complete. Digest the mixture on a steam bath for 10 minutes without stirring. Filter through No. 42 filter paper and rinse the dish with ( 1 : 19 ) dilute hydrochloric acid, scrubbing the basin with a rubber-tipped glass rod. Wash the precipitate five time with ( 1 : 19 ) hot dilute hydrochloric acid and then with hot water until free from chloride. Reserve the filterate and washings for photometric determination of residual silica.

**B-6.3.2.2** Transfer both the filter paper and precipitate to a tared platinum crucible. Ignite at a low temperature until the precipitate is free from carbonaceous matter and then heat in the muffle furnace at 1000 db 25°C to constant mass ( one hour being normally sufficient). Cool in a desiccator and weigh recording the mass as $M_1$. Moisten the contents of cold crucible, with water add five drops of dilute sulphuric acid and 10 ml of hydrofluoric acid. Evaporate to dryness on a hot plate or sand bath in a fume cupboard, taking care to avoid spurting.

**B-6.3.2.3** Heat the crucible and residue, cautiously at first, over a gas flame until the disappearance of white fumes and finally for five minutes in the furnace at 1000 ± 25°C, cool in a desiccator and weigh recording the mass as $M_2$.

**NOTE** — If the residue weight is more than 10 mg, repeat the treatment with dilute sulphuric acid and hydrofluoric acid, ignite, cool and weigh (as $M_2$). Then fuse the residue with OS g of anhydrous sodium carbonate and dissolve in 10 ml of ( 1: 4 ) dilute hydrochloric acid and transfer quantitatively to the filtrate and washings reserved in **B-6.3.2.**

**B-6.3.3** Prepare a test blank solution using the same procedure and quantities of reagents as detailed from **B-6.3.1** to **B-6.3.2** but excluding sample and dilute up the mark in a 250-ml volumetric flask.

**B-6.3.4** *Photometric Determination of Residual Silica*

**B-6.3.4.1** Boil the filtrate and washings reserved in **B-6.3.2.1** and residue to about 200 ml. Transfer to a 250-ml volumetric flask and make up to the mark with water.

**B-6.3.4.2** Transfer 10 ml aliquot of the solution prepared in **B-6.3.4.1** to a 50-ml volumetric flask. Also transfer 10 ml each of the test blank solution prepared in **B-6.3.3** to six 50-ml volumetric flasks. Add to the six flasks 0, 2, 3, 4 and 5 ml, respectively of the standard silica solution. Then to all the flasks, add 5 ml of EDTA solution, a drop of phenolphthalein solution and then add dilute ammonia solution dropwise until the colour changes to red. Then add 5 ml of ferric ammonium sulphate solution and dilute to about 30 ml with water (the *p*H of the solutions shall be 1 to 1.5 ).

**B-6.3.4.3** Add 5 ml of ammonium molybdate solution and allow to stand for 10 minutes. Then add 5 ml of oxalic acid solution followed immediately by 5 ml of ascorbic acid solution and dilute to volume to 50 ml. Allow the solution to stand for 30 minutes for development of full colour.

**B-6.3.4.4** Starting with the blank ( 0 ml) solution, transfer a suitable portion of the coloured solution one at a time to an absorption cell, record the photometric readings using the light band centred approximately at 810 nm. Prepare the calibration curve from these readings against concentration of silica in mg ( as $SiO_2$ ).

**B-6.3.4.5** Similarly, transfer a suitable aliquot of coloured test solution to the absorption cell and note the photometric readings for the test solution. Convert it to miligrams of silica by means of the calibration curve and calculate the silica content in grams in 250-ml of solution prepared in **B-6.3.3.**

### B-6.4 Calculation

Silica ( as $SiO_2$), percent by mass

where

$M$ = mass in g of the sample taken for the test in **B-6.3.1.**

$M_1$ = mass in g of crucible and contents before hydrofluoric acid treatment,

$M_2$ = mass in g of crucible and residue after hydrofluoric acid treatment, and

$M_3$ = mass in g of the silica present in 250 ml of solution prepared in **B-6.3.3.**

## B-7 DETERMINATION OF ALUMINA

### B-7.1 Outline of the Method

Aluminium is separated from the aliquot of the solution after fusing of sample with sodium carbonate as sodium aluminate and is complexed, quantitatively with EDTA by boiling with excess of the reagent. The excess EDTA is back titrated with standard zinc solution at *p*H about 5 2 using xylenol orange indicator. The aluminium EDTA complex on boiling with ammonium fluoride liberates EDTA which when titrated with standard zinc solution gives the amount of aluminium present in the solution.

### B-7.2 Reagents

**B-7.2.1** *Dilute Sulphuric Acid, 1 : 1* ( *v/v* )

Add 100 ml of concentrated sulphuric acid ( sp gravity 1.84 ) constantly stirring to 100 ml of water and cool.

**B-7.2.2** *Hydrofluoric Acid,* 40 percent ( *m/m* ) (*see IS* 10332: 1982).

**B-7.2.3** *Sodium Hydroxide Solution,* 10 percent ( *m/v* ).

**B-7.2.4** *Sodium Carbonate,* ( *see* IS 296 : 1986 ).

**B-7.2.5** *Dilute Hydrochloric Acid,* 1 : 1 ( *v/v* ).

**B-7.2.6** *Ethylene Di Amine Tetracetic Acid EDTA Solution* ( *001 M* )

Transfer 100 ml of the solution prepared in **B-6.2.6** to a 250 ml volumetric flask and dilute with water up to the mark.

**B-7.2.7** *Methyl Orange*

Dissolve 0.05 g of methyl orange in 100 ml of water.

**B-7.2.8** *Dilute Ammonia* ( *1 : 5* )

Dilute 25 ml of. concentrated ammonia (sp gravity 0.90) to 150 ml.

**B-7.2.9** *Acetate Buffer* ( *pH 52* )

Dissolve 21.5 g of sodium acetate ( $CH_3COONa.3H_2O$ ) in 300 ml of water containing 2 ml of glacial acetic acid and dilute to 1 litre.

**B-7.2.10** *Standard Aluminium Solution*

Weigh 0.529 3 g of aluminium metal (99.9 percent purity ) into a beaker containing 40 ml of dilute hydrochloric acid ( 1 : 1 ) and small drops of mercury. Heat the beaker over a water bath. Transfer the solution to a 1 000-ml volumetric flask and make up to the mark. One millilitrc of this solution is equivalent to 1.0 mg of alumina ( as $Al_2O_3$ ).

**NOTE** — Mercury will not dissolve and may be removed while transferring the solution *to* 1000-ml volumetric flask.

**B-7.2.11** *Standard Zinc Solution* ( *0.01 M* )

Dissolve 5.484 5 g of zinc acetate [ $Zn(CH_3COO)_2.2H_2O$ ] in 50-ml of water. Add 0.5-ml of glacial acetic acid and 25-ml of acetate buffer ( **B-7.2.9** ) and dilute to 1 litre in a volumetric flask. Further dilute 100-ml of this solution to 250-ml in a volumetric flask and find out the equivalent $Al_2O_3$ in g per ml of this diluted solution from the procedure given in **B-7.3.3,** taking 20-ml of standard aluminium solution ( **B-7.2.10** ).

**B-7.2,12** *Xylenol Orange Indicator*

Dissolve 0.1 g of xylenol orange in 10-ml of water containing two drops of dilute hydrochloric acid ( 1 : 1 ) .

**B-7.2.**13 *Ammonium Fluoride*

Dissolve 20 g of ammonium fluoride in 100 ml of water. Add 5 drops of xylenol orange (**B-7.2.12**) and then dilute hydrochloric acid ( 1 : 9 ) dropwise till the colour changes to yellow,

### B-7.3 Procedure

**B-7.3.1** *Solution of the Sample*

**B-7.3.1.1** Weigh accurately about 2 g of the test sample (*see* **5.1.1**) in a platinum dish. Moisten the sample with 5-ml of water. Add 3-ml of dilute sulphuric acid and 20-ml of hydrofluoric acid and evaporate slowly to dryness on sand bath or hot plate in a fume cupboard, taking care to avoid spurting. Repeat the process with 10 ml of hydrofluoric acid. Cool, add 2-ml of dilute sulphuric acid and evaporate as before. Heat the dry residue cautiously until fumes of sulphur trioxide cease. Raise the temperature to 1 000°C and ignite for five minutes. Cool and fuse the residue with 1 g of sodium carbonate and dissolve in hydrochloric acid, cool and dilute to 250 ml in a volumetric flask.

**B-7.3.1.2** Transfer 100 ml aliquot of the solution prepared in **B-7.3.1.1** to a 500-ml beaker and nearly neutralize with sodium hydroxide solution. Add with constant stirring, another 100-ml of sodium hydroxide solution containing 1 g of sodium carbonate dissolved in it. Allow to stand on a warm water bath for one hour. Finally cool to room temperature, transfer quantitatively to a 250-ral volumetric flask and dilute up to the mark with water. Mix well and allow to stand for a few minutes. Filter through a filter paper previously washed with sodium hydroxide solution, collecting the filtrate in a beaker, rinsing the beaker first with a little filtrate.

**NOTE** — Sodium carbonate is used for precipitating calcium and magnesium along with iron and titanium.

**B-7.3.1.3** Transfer a suitable aliquot from the filtrate to a 250-ml conical flask, add two drops of methyl orange indicator followed by 1:1 dilute hydrochloric acid dropwise until the colour changes from yellow to red. Add sufficient EDTA solution to provide an excess of at least 5 ml over the expected amount of alumina (1 ml of 0.025M EDTA = 1.25 mg $AI_20_3$ approximately ). Add dilute ammonia dropwise until the colour changes from red to yellow. Add 5 ml of acetate buffer and about 25 ml water and heat to boiling for 5 minutes. Cool, add 5 ml of buffer solution and titrate with standard zinc solution using 5 to 6 drops of xylenol orange indicator. The end point is indicated by a clear change from yellow to deep orange colour. Add 10 ml of ammonium fluoride solution and heat to boiling for 5 minutes. Cool, add 5 ml of buffer solution and titrate the liberated EDTA with standard zinc solution.

## B-7.4 Calculation

Alumina ( as $Al_2O_3$ ), percent by mass

where

*V* = volume in ml of standard zinc required for the titration of liberated EDTA,

*F* = equivalent $Al_20_3$ in g per ml of standard zinc solution, and

*M* = mass in g of the sample represented by 100 ml of solution taken in **B-7.3.1.2.**

## B-8 DETERMINATION OF IRON OXIDE

### B-8.1 Outline of the Method

The orange-red complex produced in acid solution of the sample (*pH* 4 to 6 ) by the addition of *o*-phenanthroline is determined photometrically at approximately 510 nm.

### B-8.2 Reagents

**B-8.2.1** *Tartaric Acid Solution,* 10 percent ( *m/v* ).

**B-8.2.2** *Hydroxylamine Hydrochloride Solution*

Dissolve 1 g of the reagent in water. Transfer the solution to a 100-ml volumetric flask, dilute to the mark and mix well.

**B-8.2.3** *Ortho-Phenanthroline Solution*

Dissolve 1 g of *o*-phenanthroline monohydrate in 90-ml of water with gentle heating and constant stirring. Cool and dilute to 100 ml.

**B-8.2.4** *Acetate Buffer*

Dissolve 21.5 g of sodium acetate ( $CH_3COONa. 3H_2O$) in 300-ml of water containing 2 ml of glacial acetic acid and dilute to 1 litre.

**B-8.2.5** *Standard Iron Solution*

Dissolve 0.2 g of pure iron in concentrated hydrochloric acid. Dilute the solution to about 100 ml. Transfer to a 100-ml volumetric flask, dilute to the mark and mix well. Dilute 10 ml of this solution to 1 000-ml in a volumetric flask. One millilitre of this diluted solution is equivalent to 0.02 mg of iron (as Fe).

### B-8.3 Procedure

**B-8.3.1** Transfer a S-ml aliquot of the solution prepared in **B-7.3.1.1** to a 100-ml volumetric flask. Also transfer 0, 1, 2, 4, 6, 8 and 10 ml each of the standard iron solution to six separate 100-ml volumetric flasks. Then add to each flask, 2 ml of tartaric acid solution followed by 2 ml of hydroxylamine hydrochloride solution. Stir well and add 5 ml of o-phenanthroline solution and 10 ml of acetate buffer solution. Allow to stand for 1S minutes and dilute up to the mark with water.

**B-8.3.2** Starting with the blank (0 ml) solution, transfer a suitable portion of the coloured solution, one at a time, to an absorption cell; record the photometric readings using the light band. centred approximately at 510 nm, prepare the calibration curve from these readings against concentration of iron ( as Fe ).

**B-8.3.3** Similarly transfer a suitable aliquot of the coloured test solution to the absorption cell and note the photometric reading for the test solution. Convert it to milligrams of iron by means of the calibration curve and calculate the iron oxide content in the aliquot taken in **B-8.3.l.**

**B-8.4** Calculation

Ferric oxide ( as $Fe_2O_3$),

percent by mass

where

$M$ = mass in mg of iron ( as Fe ) found in aliquot, and

$M_1$ = mass in g of the sample represented by the aliquot taken in **B-8.3.1.**

## B-9 DETERMINATION OF TITANIUM DIOXIDE

### B-9.1 Outline of the Method

The yellow coloured complex, produced in acid solution of the sample by the addition of hydrogen peroxide, is determined photometrically at approximately 410 nm.

### B-9.2 Reagents

**B-9.2.1** *Dilute Sulphuric Acid,* 1 : 1 ( *v/v* ).

**B-9.2.2** *Dilute Phosphoric Acid*

Add 400 ml of phosphoric acid ( sp gravity 1 75 ) to 500 ml of water, cool and dilute to 1 litre.

**B-9.2.3** *Hydrogen Peroxide,* 20 volumes ( *see* IS 2080 : 1980).

**B-9.2.4** *Standard Titanium Dioxide Solution*

Weigh accurately 3*7016 g of potassium titanyl oxylate [ $K_2TiO_2 ( C_2 0_4 )_2.2H_2O$ ]. Transfer to a boiling flask, add 8 g of ammonium sulphate and 100 ml of concentrated sulphuric acid. Gradually heat the mixture to boiling and boil for 10 minutes. Cool, pour the solution into 750 ml of water, and dilute to 1 litre in a volumetric flask. One millilitre of this solution is equivalent to 0.5 mg of titanium dioxide ( as $TiO_2$).

### B-9.3 Procedure

Transfer 25 ml of the prepared solution **B-7.3.1** to a 100-ml volumetric flask. Add 2.5 ml of dilute sulphuric acid, 5 ml of dilute phosphoric acid and 5 ml of hydrogen peroxide. Make up to the mark with water and mix.

**B-9.3.1** *Calibration Curve*

Transfer 25 ml each of the blank solution to six 100-ml volumetric flasks and add 0, 1, 2, 3, 4 and 5 ml, respectively of the standard titanium dioxide solution, using the same quantity of reagents and procedure as in **B-9.3.** Make up the solutions up to the mark. Take the required aliquots of these solutions in the absorption cell, one at a time, and plot a curve showing optical densities of solutions at 410 mm against the amount of titanium dioxide in milligrams present in each.

NOTE — The test blank solution shall be prepared by using the same reagents as given in **B-7.3.1.1** but without the material and dilute the solution with water to 250 ml in a volumetric flask.

**B-9.3.2** Next take appropriate aliquot of the solution prepared in **B-9.3** in the absorption cell and measure its optical density at 410 nm and note from the calibration curve the amount of titanium dioxide in milligrams present in it.

### B-9.4 Calculation

Titanium dioxide ( as $TiO_2$ ),

percent by mass

where

$M_1$ = mass in mg of titanium dioxide content present in the aliquot taken for the test ( **B-9.3.2** ), and

$M$ = mass in g of the material present in the aliquot taken for the test ( **B-9.3** ).

## B-10 DETERMINATION OF WATER OF PLASTICITY

**B-10.1** Outline of the Method

The water content of bentonite at the point of maximum workability is determined. This when expressed as a percentage of oven dried bentonite gives the values for water of plasticity.

**B-10.2** Apparatus

**B-10.2.1** *Spatula*

**B-10.2.2** *Porcelain Basin,* conforming to IS 2837 ( Part 2 ): 1977.

**B-10.2.3** *Burette,* 50 ml capacity (*see* IS I997 : 1982).

**B-10.2.4** *Balance,* accurate up to 0.01 g.

**B-10.2.5** *Test Sieve,* 250 micron IS Sieve.

### B-10.3 Procedure

**B-10.3.1** Dry thoroughly 500 g of bentonite at 105 ±2°C and crush to pass through 250 micron IS Sieve. Work up the sieved bentonite with water, by adding small quantities of water from the burette, to a soft plastic consistency by thoroughly wedging and kneading by hand. Allow it to age for 24 hours. During the period of ageing, it should be covered by wet cloth. After 24 hours, knead the plastic mass once again and pug it well by further small additions of water working consistency for either extrusion or moulding into bars is obtained. Take three equal portions of the plastic mass, at this consistency from different portions, the edges and corners rounded to avoid any handling loss. Weigh immediately to the nearest 0.01 g.

**B-10.3.2** Dry the three masses at room temperature for 24 hours and then at 105± 2°C for 24 hours. Cool in a desiccator and weigh to nearest 0.01 g.

### B-10.4 Calculation

Water of plasticity, percent

where

$A$ = mass in g of the plastic mass, and

$B$ = mass in g of the dried mass.

## B-ll TEST FOR SWELLING POWER

### B-ll.l Procedure

Add 2 0 g of the test sample ( *see* **5.1.8** ) in 20 approximately equal portions at intervals of 2 minutes, to 100 ml of solution of sodium lauryl sulphate ( 1 percent *m/v* ) contained in 100 ml measuring cylinder. Note the volume after the addition of the material. Then allow the material to swell for 24 hours. Note the volume after 24 hours. The difference in volume after 24 hours shall be reported as the swelling power of the sample.

## B-12 DETERMINATION OF CALCIUM OXIDE AND MAGNESIUM OXIDE

### B-12.1 Outline of the Method

In an aliquot of the sample solution, metallic acid inpurities of group element ( $R_20_5$ ) are marked by triethanelamine, and calcium and magnesium are titrated with EDTA solution at *p*H 10 using Eriochrome black-T indicator. In another aliquot, magnesium is precipated out at *p*H 12 in the presence of triethanolamine and calcium is titrated with EDTA using Patton and Reader ( P & R ) indicator. Subtracting the titre value of calcium from that of calcium and magnesium, the titre value for magnesium is obtained.

### B-12.2 Reagents

**B-12.2.1** *Concentrated Nitric Acid, see* IS 284 : 1976.

**B-12.2.2** *Perchloric Acid,* 70 percent ( *m/m* ).

**B-12.2.3** *Hydrochloric Acid,* 40 percent (*m/m* ).

**B-12.2.4** *Concentrated Hydrochloric Acid, see* IS 265: 1976.

**B-12.2.5** *Trlethanolamlde Solution,* 30 percent (*v/v*)

Dilue 60 ml of triethanolamine to 200 ml.

**B-12.2.6** *Sodium Hydroxide Solution* ( 5N approximately )

Dissolve 100 g of sodium hydroxide in water, cool and dilute to 500 ml and store in a polyethylene bottle.

**B-12.2.7** *Patton and Reader* ( *P & R* ) *Indicator*

Mix by grinding together 0.1 g of P & R indicator and 30 g of sodium chloride. About 0.2 g of the mixture is required for each titration.

**B-12.2.8** *Ethylene Diamine Tetracetic Acid* ( *EDT ) Solution* 0.01 M approximately.

Dissolve 3.8 g of disodium ethylenediamine tetracetate dihydrate ($C_{10}H_{14}N_2.Na_2O_3.H_2O$ ) in water and dilute to 1 litre. Standardized against standard calcium solution ( *see* **B-12.2.9** ) using P & R indicator and against the standard magnesium solution (*see* **B-12.2.10** ) using Erichrome black-T indicator.

**B-12.2.9** *Standard Calcium Solution*

Dissolve 1.784 8 g of calcium carbonate dried at 150°C in a slight excess of hydrochloric acid, boil to. expel carbon dioxide, cool and dilute to 1 litre. One millilitre of this solution is equivalent to 1 mg of calcium oxide ( as CaO ).

**B-12.2.10** *Standard Magnesium Solution*

Wash a piece of magnesium in dilute hydrochloric acid to remove any oxide film, then with water and dry it with ethanol followed by ether. Dissolve 0.603 2 g of the metal in a slight excess of dilute hydrochloric acid, cool and dilute to 1 litre. One millilitre of this solution is equivalent to 1 mg of magnesium oxide [ as ( Mg O)].

**B-12.2.11** *Ammonia-Ammonium Chloride Buffer Solution* (*p*H — *10*)

Dissolve 57.5 g of ammonium chloride in 250 ml of water, and 570 ml of ammonia ( sp gravity 0.88 ) and dilute to 1 litre and mix thoroughly.

**B-12.2.12** *Erichrome Black-T Indicator Solution*

Dissolve 0.4 g of the sodium salt of erichrome black-T in a mixture of 20 ml of ethanol and 30 ml of triethanolamine.

### B-12.3 Procedure

**B-12.3.1** *Solution of the Sample*

Weigh accurately about 1 g of the test sample ( *see* **5.1.1** ) in a platinum dish. Add 5 ml each of concentrated nitric acid and perchloric acid and about 10 ml of hydrofluoric acid. Cover the dish with a lid. Allow the reaction to proceed for at least 15 minutes, then remove the lid and evaporate the mixture to dryness on a sand bath in a fume cupboard, taking care to avoid spurting. Cool, add 5 ml of perchloric acid, rinse the lid and sides of the dish with water and again evaporate carefully to dryness. To the cool, dry residue, add 2 ml of concentrated hydrochloric acid and about 15 ml of water. Digest on a steam bath for 10 minutes, cool, filter if necessary and dilute the solution with water to 250 ml in a volumetric flask. Store the solution in a polyethylene bottle.

**B-12.3.1.1** *Determination of calcium oxide*

Pipette out 50 ml of the solution into a 250 ml conical flask. Add 10 ml triethanolamine solution and 5 ml of sodium hydroxide solution dropwise while shaking the solution and then add 0.2 g of P & R indicator. Titrate immediately with EDTA solution until the colour changes from pink to blue.

**NOTE** — If sharp end-point is not obtained, add 2 ml of potassium cyanide ( 10 percent *m/v*) after the addition of triethanolamine.

*Calculation*

Calcium oxide ( as CaO ), percent by

mass

where

$V$ = volume in ml of EDTA solution required for titration,

$F$ = CaO equivalent in g/ml of the EDTA solution, and

$M$ = mass in g of the sample represented by the aliquot taken.

**B-12.3.1.2** *Determination of magnessium oxide*

Pipette out 50 ml of solution reserved in **B-12.3.1** into a 250-ml conical flask. Add 10 ml of triethanolamine solution. Add 15 ml of ammonia-ammonium chloride buffer solution. Add 4 to 5 drops of ErichTome black-T indicator solution and immediately titrate with the EDTA solution until the colour changes from wire red to blue.

*Calculation*

Magnesium oxide ( as MgO ), percent

by mass

$M$

where

$V_1$ = volume in ml of EDTA solution required for titration of total calcium and magnesium required in **B-12.3.1.2,**

$V$ = volume in ml of the EDTA solution required for titration in **B-12.3.1.1.**

$F$ = MgO equivalent in g/ml of the EDTA solution, and

$M$ = mass in g of the sample represented by the aliquot taken.

## B-13 DETERMINATION OF APPARENT VISCOSITY

### B-13.1 Apparatus

**B-13.**1.1 *Direct Indicating Torsion Viscometer*

The instrument is based on the principle that the torque exerted by a liquid is directly proportional to its viscosity. It consists of two cylinders. Mud ( suspended material ) is contained in the annular space between two cylinders. The outer cylinder or rotor sleeve is connected to a motor and is driven at a constant rotational velocity. The rotation of the rotor sleeve in the mud ( suspended material) produces a torque on the inner cylinder or bob. A torsion spring restrains the movement. A dial attached to the bob indicates displacement of the bob. The instrument constants have been so adjusted that viscosity and yield point are obtained by using readings from rotor sleeve speeds of 600 rev/min The apparent viscosity in centipoise equals the 600 rev/min reading divided by 2.

**B-13.1.2** ***Electric Stirrer***

A high speed laboratory electric stirrer having a minimum speed of 5 000 rev/min with corrugated impeller.

### B-13.2 Procedure

Prepare a suspension of 22.5 g of the dried material in 350 ml of water with the electric stirrer, and age for 24 hours at a temperature of 30 ± 2°C. Place a recently stirred sample of the aged suspension in a suitable container and immerse to rotor sleeve exactly to the scribed line. With the sleeve rotating at 600 rev/min, wait for the dial reading to reach a steady valve [ the time required is dependent on the mud ( suspended material ) characteristics ]. Record the dial reading for 600 rev/min.

### B-13.3 Calculation

Apparent viscosity at 30°C in centipoise

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